THE MINERALOGICAL MAGAZINE
AND
JOURNAL OF
THE MINERALOGICAL SOCIETY

No. 172 March, 1941 Vol. XXVI

On sulphatic cancrinite and analcime (eudnophite) from Loch Borolan, Assynt.

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[Read June 6, 1940; communicated by Prof. C. E. Tilley, F.R.S.]

THE minerals to be described in this paper were collected, in conjunction with W. E. Fraser, who was carrying out a detailed study of the borolanite, from the road-metal quarry, opened in 1938, at Allt a' Mhuillin, Loch Borolan, Assynt, Scotland. They occur in pegmatitic patches and veins in borolanite.

I. Sulphatic Cancrinite.

This mineral is one of the chief constituents of the pegmatites. It forms large cleavable masses, associated with orthoclase, melanite, and a little dark mica. Calcite, sphene, zircon, and orthite are present as accessories. It often fills interstices between crystals of orthoclase. A preliminary examination has indicated that it is a common constituent of the spots in some types of borolanite, and further work will have to be done to determine its importance in this connexion. This is the first definite record of the occurrence of cancrinite in Britain.

The cancrinite is light blue to light greyish-blue in colour. It has a density of 2·42, and its hardness is 5. It effervesces in hydrochloric acid, and gelatinous silica is formed on heating. It is uniaxial negative, but occasionally it gives a biaxial interference-figure with a very small axial angle (2° or 3°). The highest refractive index is much lower and the birefringence much weaker than that of normal cancrinite. For sodium-light: ω 1·502, ϵ 1·497, $\omega - \epsilon$ 0·005.

The results of a chemical analysis of pure material are given in table I,

together with the molecular ratios. The atomic proportions calculated on the basis of Al+Si = 12 are included in table II.

TABLE I. Chemical analyses and molecular ratios of sulphatic cancrinite.

	I.	II.	III.	IV.	I.	II.	III.	IV.
SiO_2	34·76	33·70	34·78	35·29	0·579	0·561	0·579	0·588
Al_2O_3	30·81	29·40	28·77	28·79	0·302	0·289	0·282	0·282
CaO	3·87	4·18	1·27	1·49	0·069	0·075	0·023	0·027
SrO	0·32	0·08	—	—	0·003	0·001	—	—
Na_2O	18·90	18·52	15·59	15·65	0·305	0·299	0·252	0·252
K_2O	1·29	1·45	4·90	4·15	0·014	0·015	0·052	0·044
H_2O+	2·30	4·24	7·01	7·62	0·139	0·275	0·389	0·423
H_2O-	0·20	0·72						
CO_2	1·90	3·18	0·89	1·01	0·043	0·072	0·020	0·023
SO_3	5·93	4·65	6·25	5·76	0·074	0·058	0·078	0·072
Total	100·28	100·19	99·68	100·05				

I. Allt a' Mhuillin, Loch Borolan, Assynt, Scotland. Analyst, F. H. Stewart.
II. Beaver Creek, Colorado. Analyst, G. Steiger in Larsen and Steiger (1916). Includes TiO_2 0·07.
III and IV. Ilmen Mts., southern Ural. Analyst, Y. V. Morachevsky in A. N. Zavaritzky (1929). III includes Fe_2O_3 0·10, MgO 0·12; IV includes Fe_2O_3 0·19, MgO 0·10.

In 1893 Horne and Teall, in a paper on borolanite (pp. 174–175), state:

'A peculiar blue substance occurs wedged in between the large individuals of orthoclase in certain veins and is found also as a constituent of some of the white spots. It shows aggregate polarisation, and is decomposed by hydrochloric and sulphuric acids, with the separation of gelatinous silica and the evolution of bubbles.

After adding water to the hydrochloric acid solution and evaporating slowly, salt and gypsum crystals are developed—the former in great abundance. A partial analysis was made on about half a gramme of this substance, with the following result:—

SiO_2.	Al_2O_3.	CaO.	K_2O.	Na_2O.	SO_3.	Total.	Sp. gr.
36·1	28·4	3·2	1·8	16·2	5·9	91·6	2·41–2·43

Water and carbonic acid are present, but were not determined. The reaction of this substance with acid is suggestive of cancrinite, but the occurrence of sulphuric acid points to the conclusion that it is an alteration product after a mineral of the sodalite group.'

The blue mineral described by Horne and Teall is obviously the same as the sulphatic cancrinite from Allt a' Mhuillin. The analyses correspond fairly well, though the Allt a' Mhuillin one has about 3 % more $Na_2O+K_2O+CaO+SrO$. $SiO_2+Al_2O_3$ is about the same. The material analysed by Horne and Teall was probably impure, as it is very unlikely that the 8 % needed to complete the analysis consisted solely of water and CO_2.

In the Geological Survey Memoir on the 'Geological structure of the north-west Highlands of Scotland' (1907, pp. 445–446), a white or pale blue alteration product 'presumably after nepheline', is mentioned,

occurring in a pegmatite immediately east of Allt a' Mhuillin. An analysis of the white substance is given, and this shows 1·67 % SO_3 and 0·57 % CO_2. It is remarked that 'The presence of sulphuric acid is remarkable, and suggests the presence in the original rock of a mineral of the Hauyn group. Chlorine was looked for but not found.'

Apart from three other records of sulphatic cancrinite, cancrinite analyses usually show that no SO_3 is present. A few have very small amounts of SO_3. A cancrinite from Siksjöberg, Sweden,[1] contained 0·54 %, one from Miask, Ural,[1] 0·32 %, from Dôdo, Korea (Kôzu, 1931), 0·08 %, and from Iron Hill, Colorado (Larsen and Foshag, 1926) 0·03 %. In 1916 Larsen and Steiger described a sulphatic cancrinite, with 4·65 % SO_3, from Beaver Creek, Colorado; it was nearly colourless and occurred in an altered uncompahgrite associated with apatite, perovskite, and melilite. An analysis was made by Steiger, and this is quoted in table I above. In 1929 Zavaritzky described a bluish sulphatic cancrinite from the Ilmen Mountains, southern Ural, which occurred as small nests and veinlets in large crystals of nepheline in pegmatite veins in nepheline-syenite. He gives two analyses by Morachevsky which are quoted in table I, and which show 6·25 and 5·76 % SO_3. In 1931 Belyankin described a blue sulphatic cancrinite, very similar to that of the Ilmen Mountains, from Vishnevy Mountains, southern Ural. It occurred in a felspar quarry, and was associated with nepheline, aegerine, dark mica, ilmenite, magnetite, sphene, and zircon. He gives an analysis,[2] but estimates that the analysed material contained 10 % of scolecite and gibbsite as impurities. He suggests the name vishnevite (wischnewite) for the sulphatic end-member of the series. In 1911 Lacroix described a mineral from the nepheline-aegerine-syenite of the Los Islands, West Africa, which he named losite, and which he said was a member of the cancrinite group with low birefringence. It is fibrous and elongated parallel to the vertical axis, and occurs replacing nepheline. It is uniaxial negative, $\omega - \epsilon$ 0·011–0·012. The lowest refractive index is about 1·49–1·50. 'En raison de la biréfringence donnée plus haut, l'indice maximum peut être estimé à environ 1·51.' It gelatinizes readily with acid. The properties of this mineral indicate that it is a sulphatic cancrinite, but unfortunately it has not been analysed. In 1912 Lacroix described another occurrence of losite in a nepheline-gneiss from Madagascar.

Table II shows the atomic proportions, calculated on the basis Si+Al

[1] Analyses are given in C. Hintze, Handbuch der Mineralogie, 1897, vol. 2, p. 881.

[2] This analysis is not quoted, because of the presence of impurities, and because the summation of the analysis, as printed, is not correct.

= 12, from the analyses of a synthetic lime-free cancrinite, two normal cancrinites (figures taken from Berman, 1937), and the sulphatic cancrinites.

TABLE II. Atomic proportions of cancrinite and sulphatic cancrinite, taking Si+Al = 12.

	Synthetic (Friedel).	Litchfield, Maine.	Iron Hill, Colorado.	Beaver Ck., Colorado.	Allt a' Mhuillin.	Ilmen Mts.	Ilmen Mts.
				II.	I.	III.	IV.
Si	5·98	6·10	5·86	5·92	5·87	6·08	6·12
Al	6·02	5·90	6·14	6·08	6·13	5·92	5·88
Ca	—	0·93	1·61	0·79	0·70	0·24	0·28
Sr	—	—	—	0·01	0·03	—	—
Na	7·17	6·38	5·19	6·30	6·19	5·28	5·26
K	0·50	0·02	0·11	0·32	0·28	1·09	0·92
O	23·79	23·69	23·59	23·69	23·71	23·43	23·43
H_2O ...	2·30	2·16	2·68	2·90	1·41	4·08	4·40
CO_3 ...	1·05	1·51	1·61	0·76	0·44	0·21	0·24
SO_4 ...	—	—	—	0·61	0·75	0·82	0·75

The general formula, $W_{7-8}Z_{12}O_{24}\cdot N\cdot sH_2O$, is given by Berman (1937) for the cancrinite group, where W = Na, Ca, K, with Na and Ca predominating, Z = Si : Al = 1 : 1, and N = CO_3, SO_4. The four analyses of sulphatic cancrinite would correspond to the general formula:

$$(Na,K,Ca,Sr)_{6-8}(Si,Al)_{12}O_{24}(SO_4,CO_3)_{1-2}\cdot 1\text{–}5H_2O.$$

The water-content varies greatly, and in the Allt a' Mhuillin mineral it is very low. The Ilmen Mountains sulphatic cancrinite is very rich in water and low in oxygen.

The properties of the sulphatic cancrinites and of some normal cancrinites are given in table III, with the weight percentage of SO_3, and SO_3 as molecular per cent. of (SO_3+CO_2).

TABLE III. Optical data of cancrinite and sulphatic cancrinite.

Locality.	ω.	ϵ.	$\omega-\epsilon$.	Sp. gr.	Wt. % SO_3.	SO_3 mol. % of (SO_3+CO_2).
Litchfield, Maine	1·522	1·499	0·028	2·448	—	—
Miask, Ural ...	1·5244	1·4955	0·0289	2·453	—	—
Korea	1·5238	1·5015	0·0223	2·44	0·08	0·74
Iron Hill, Colo. ...	1·524	1·501	0·023	2·51	0·03	0·26
Beaver Ck., Colo....	1·509	1·500	0·009	2·443	4·65	44·56
Allt a' Mhuillin ...	1·502	1·497	0·005	2·423	5·93	63·18
Ilmen Mts. ...	1·493—1·492		0·0007	2·35	6·25—5·76	79·43—75·84

Figures for Litchfield and Miask were taken from C. Hintze, Handbuch der Mineralogie, 1897, vol. 2, p. 877.

From the data in table III it can be seen that the birefringence de-

creases from 0·028[1] in cancrinite to nearly zero in the sulphatic cancrinite from the Ilmen Mountains. Larsen and Steiger (1916, p. 334), discussing the sulphatic cancrinite from Beaver Creek, state that 'Sulphatic cancrinite has a much lower birefringence than cancrinite and it is not unlikely that a member of the group somewhat richer in sulphate has zero birefringence and that the pure sulphate member is optically negative'. (By 'negative' the authors must mean positive, as all *normal* cancrinites are negative.) The more recent evidence supports these views.

Of the two refractive indices the higher (ω) is the one most affected. It falls fairly rapidly from 1·524[1] (0 % SO_3) to 1·493 (79·43 % SO_3, as mol. % of SO_3+CO_2). The changes of the lower refractive index (ϵ) are rather unreliable, as all the values shown come within the range of Dana's values for cancrinite. However, in the sulphatic members ϵ decreases with increase of SO_3.

The optical properties may be influenced to some extent by the water-content, which is very variable. The remarkably low density of the sulphatic cancrinite from the Ilmen Mountains is probably due to its high water-content (4·08–4·40 atomic proportions).

II. Analcime.

Analcime was found in a patch about a foot in width near the centre of the quarry, associated with orthoclase, melanite, sphene, and orthite. It formed a large white cleaved mass, with a vitreous lustre. There are three well-developed cleavages at right angles to each other.

It has a hardness of just over 5, and a density of 2·268. It gelatinizes with acid, and fuses readily to a colourless glass. A chemical analysis is given in table IV with the molecular values and the atomic proportions calculated on the basis O = 14.

Table IV. Chemical analysis of analcime from Allt a' Mhuillin, Loch Borolan, Assynt, Scotland. Analyst, F. H. Stewart.

		Molecular ratios.		No. of metal atoms to 14 oxygens.		
SiO_2	52·89	0·8804	Si	...	3·909	
Al_2O_3	24·63	0·2417	Al	...	2·146	
CaO	0·19	0·0034	Ca	...	0·015	
Na_2O	13·31	0·2147	Na	...	1·906	1·99
K_2O	0·73	0·0078	K	...	0·069	
H_2O+	7·66	0·4412	H	...	3·918	
H_2O-	0·29		O	...	14·000	
	99·70					

[1] The properties of normal cancrinites vary. This is the value given by Larsen and Berman (1934, p. 78). Dana's values are ω 1·515—1·524, ϵ 1·491—1·502, sp. gr. 2·42—2·5 (Textbook of mineralogy, 4th edition, 1932).

This analysis corresponds to the formula $(Na,K,Ca)_2Al_2Si_4O_{12}.2H_2O$. There is a little replacement of silicon by aluminium.

The refractive index for sodium-light is 1·486—1·488 and the mineral shows a very definite birefringence, up to 0·002. It usually gives a biaxial interference-figure, optically negative, and occasionally a uniaxial figure. The birefringence varies, and some small patches are isotropic. The mineral nearly always shows lamellar twinning, which is best developed parallel to one of the cleavages; this cleavage will be called the 'vertical' cleavage, so as to define a direction. The optic axial angle varies from 0° to 40°. The optic axial plane is generally only slightly inclined to the 'vertical' cleavage, but some cleavage flakes show a remarkably wide variation in its position. For example, in one cleavage flake roughly normal to α the position of the optic axial plane varies quite irregularly up to nearly 90° from the 'vertical' cleavage about the axis of the microscope, and up to 20° about the horizontal axes. Within a lateral motion of 0·3 mm. in one part of the slide, the axial angle decreases from 35° to 0° and then opens out at right angles and increases to 30°.

The best developed twin-lamellae are parallel to the 'vertical' cleavage, and usually almost parallel to the axial plane. They are seen as fine lines, often discontinuous, in cleavage flakes roughly normal to α and γ. Only very occasionally are the lamellae large enough for determination of the extinction-angles, and in these cases the extinction is usually symmetrical, up to 10° on either side of the 'vertical' cleavage. Twin-lamellae are usually seen parallel to the other two cleavages, but these are not nearly so well defined, and they are often irregular and spindle-shaped.

Some sections show dodecahedral twinning, as a very fine set of lamellae at 45° to the cleavages, but these are rare. Another type of dodecahedral twinning has been seen in some sections normal to γ. Here large untwinned lamellae up to 5 mm. across with straight extinction alternate with lamellae which show twinning on a minute scale parallel to the two cleavages. The twinned lamellae have in general a slightly oblique extinction of 1° to 3°. The boundaries between these two types of lamellae are sometimes sharp and straight, but often very irregular.

Birefringent twinned analcimes have been described from many localities, and probably the most noteworthy of these is the analcime from Låven, in south Norway, which was first described by Weibye in 1850, and which he called eudnophite. He gave measurements of interfacial angles of crystals which he said were orthorhombic and which showed the prism, brachypinakoid, and macrodome, but these were proved later

to be measurements of felspar crystals (Brögger, 1890, pp. 565-566). The rest of his material was shown by Brögger to be a birefringent variety of analcime showing lamellar twinning. Brögger gave a detailed description of the Låven analcime, and described and figured orientated sections. The mineral occurred as white, semi-transparent icositetrahedra, enclosed in felspar. It showed good cubic cleavage. The most important twin-lamellae were parallel to the three cubic cleavages, but subordinate dodecahedral lamellae were also present. The birefringence varied, some parts being isotropic. The mineral was usually biaxial, but occasionally uniaxial. These anomalies described by Brögger are exactly similar to those of the Allt a' Mhuillin analcime. Brögger also describes small icositetrahedral crystals, in which the periphery shows a zonal arrangement parallel to the icositetrahedral edges, and the nucleus shows lamellar twinning. This zonal arrangement has not been seen in the analcime from Allt a' Mhuillin, which does not show crystal faces. Sector twinning, which has often been recorded in analcimes,[1] is also absent.

The optical anomalies shown by analcime have never been satisfactorily explained. Klein (1897) described the effects of heating and loss of water on the optical properties, and he found that when analcime was heated in a dry atmosphere the birefringence increased with loss of water. When the mineral was heated to 250° in the presence of water vapour it usually became isotropic, but on cooling it became birefringent again. Analyses of the Låven eudnophite, as Brögger pointed out, show no deficiency in water-content.

The fact that the optical properties of the Allt a' Mhuillin mineral vary so much, and so irregularly, indicates that the anomalies might be due to some sort of strain during crystallization. However, this problem must remain unsolved at present.

The writer is indebted to Professor C. E. Tilley for valuable help and criticism. The material was collected while the writer was a Kilgour Scholar at Aberdeen University, and the determinative work was carried out at Cambridge during the tenure of a Carnegie Research Scholarship.

Summary.

A sulphatic cancrinite, with 5·93 % SO_3, is described from Loch Borolan, Assynt, Scotland, and compared with three other sulphatic cancrinites previously described, and with normal cancrinites. They form an

[1] Cyclopean Islands and Kerguelen (Arzruni and Koch, 1881), Japan (Jimbō, 1915), Texas (Lonsdale, 1928), &c.

isomorphous series, in which SO_3 replaces CO_2, and the sulphatic end-member has not yet been recorded. The influence of the SO_3 content on the optical properties is discussed. It is shown that the higher refractive index (ω) and the birefringence decrease fairly rapidly with increasing SO_3. The mineral probably changes from negative to isotropic and then to positive, with increasing SO_3 content.

A birefringent variety of analcime occurs in a pegmatitic patch in the borolanite, Loch Borolan. It is optically negative and usually biaxial. It shows twin-lamellae parallel to the cubic cleavages, and occasional subordinate dodecahedral lamellae. The optical anomalies are very similar to those of eudnophite, and it is suggested that they may be due to strain during crystallization.

References.

1850, Weibye (P. C.). Ann. Phys. Chem. (Poggendorff), vol. 79, p. 303.
1881, Arzruni (A.) and Koch (S.). Zeits. Kryst. Min., vol. 5, pp. 483–489.
1890, Brögger (W. C.). Zeits. Kryst. Min., vol. 16, pp. 565–585.
1893, Horne (J.) and Teall (J. J. H.). Trans. Roy. Soc. Edinburgh, vol. 37 (pt. 1 for 1891–2), pp. 163–178.
1897, Klein (C.). Sitzungsber. Akad. Wiss. Berlin, pp. 290–354.
1907, Teall, Peach, Horne, *et al.* The geological structure of the north-west Highlands of Scotland. Mem. Geol. Surv. Great Britain.
1911, Lacroix (A.). Nouv. Arch. Muséum Hist. Nat. Paris, ser. 5, vol. 3, p. 37.
1912, — Compt. Rend. Acad. Sci. Paris, vol. 155, p. 1125.
1915, Jimbō (K.). Beitr. Min. Japan, no. 5, pp. 295–296. [M.A. **1**–156.]
1916, Larsen (E. S.) and Steiger (G.). Amer. Journ. Sci., ser. 5, vol. 42, pp. 332–334. [M.A. **1**–256.]
1926, Larsen (E. S.) and Foshag (W. F.). Amer. Min., vol. 11, pp. 300–303. [M.A. **3**–452.]
1928, Lonsdale (J. T.). Amer. Min., vol. 13, pp. 449–450. [M.A. **4**–96.]
1929, Zavaritzky (A. N.). Mém. Soc. Russe Min., ser. 2, vol. 58, pp. 201–207. [M.A. **4**–379.]
1931, Belyankin (D. S.). Centr. Min., Abt. A., pp. 190–196. [M.A. **4**–499.]
1931, Kôzu (S.). Japanese Journ. Geol. Geogr., vol. 9, Abstracts, pp. 1–2. [M.A. **5**–48.]
1934, Larsen (E. S.) and Berman (H.). The microscopic determination of the nonopaque minerals. Bull. U.S. Geol. Surv., no. 848, 2nd edition. [M.A. **5**–481.]
1937, Berman (H.). Amer. Min., vol. 22, p. 369.

The selenium and tellurium contents of sulphur from Krisuvik, Iceland.[1]

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[Read June 6, 1940.]

THE deposits of sulphur at Krisuvik, Iceland, have long been known. They were described geologically in detail by Vincent[2] who also discussed their probable origin, but gave no chemical analyses other than rough determinations of the percentages of sulphur and matrix in various samples. From the flame spectrum Vincent thought that thallium might be present.

Apart from popular descriptions by travellers and casual references in guide-books, little attention appears to have been given in literature to these deposits in more recent years. Sporadic attempts at working the sulphur economically were made by the Danish Government during the eighteenth century and later by a Danish merchant round 1832. These were abandoned, however, owing to cost of transport. There is little likelihood of their renewal in the immediate future unless the deposits are found to contain some other element(s) of sufficient market value.

In view of the increasing commercial applications of selenium and tellurium it seemed of interest to ascertain if either or both of these elements were present in appreciable quantities in the Krisuvik deposits. The results were not encouraging but may be worth placing on record.

About 1 kilogram of the crude mineral was obtained through the good offices of Mr. Stefan Stefánsson of Reykjavik and 50 gram lots were boiled stepwise under reflux with carbon disulphide until all soluble matter had been extracted. The insoluble siliceous residue (3·2 %) and the soluble portion recovered by distillation were examined spectrographically with a Hilger quartz spectrometer.

Both portions showed lines due to selenium and tellurium, whilst the insoluble residue gave also strong lines due to titanium, weaker nickel lines, and lines due to iron, calcium, and barium.

The presence of the nickel was subsequently confirmed by the

[1] A preliminary note appeared in Nature, London, 1939, vol. 143, p. 762.
[2] C. W. Vincent, Chem. News, 1873, vol. 27, pp. 111, 126.

dimethylglyoxime test, that of titanium by the peroxide test, the metal being estimated colorimetrically by the same reaction. Tellurium was confirmed qualitatively by precipitation as minute rods with quinol and concentrated sulphuric acid, and examination under the microscope.[1] It was quantitatively determined by digestion of the extracted sulphur and of the insoluble residue respectively with concentrated nitric acid, extraction of the selenium and tellurium dioxides with potash, precipitation of the former from the acidified solution with sodium sulphite, and colorimetric comparison of the filtered solution after reduction with mercurous chloride against a suitable standard.[2]

Selenium was confirmed qualitatively by the thio-urea test[3] and subsequently estimated by oxidation of the extracted sulphur and residue respectively with nitric acid to selenious acid. Excess nitric acid was removed by evaporation with hydrochloric acid to prevent nitration of the pyrrole, which latter was then added, together with orthophosphoric acid and ferric chloride. The blue colour was compared with a standard.[4] The tellurium was twenty times as abundant as the selenium in both the extracted sulphur and the insoluble residue.

The results expressed in parts per million were as follows:

	Extracted sulphur. p.p.m.	Insoluble residue. p.p.m.	Crude mineral. p.p.m.
Titanium	—	3750	120
Selenium	12·5	200	18·9
Tellurium	250	4000	378

[1] P. C. Putnam, E. J. Roberts, and D. H. Selchow, Amer. Journ. Sci., 1928, ser. 5, vol. 15, p. 253.

[2] G. G. Pierson, Ind. Eng. Chem., Anal. Edit., 1934, vol. 6, p. 437.

[3] M. H. Evans, Amer. Min., 1937, vol. 22, p. 1128.

[4] R. Berg and M. Teitelbaum, Mikrochem. Emich Festschr., 1930, p. 23.

Optical properties of olivines from Ubekendt Island, west Greenland.

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[Read November 7, 1940.]

THE olivines, which are the subject of this note, were obtained from a series of basaltic lavas and dikes on Ubekendt island, which lies off the west coast of Greenland in latitude 71° N., longitude 54° W.

The optical properties of the olivines were investigated in order to determine any range in composition; such variations might have been expected in the very thick lava series. From this aspect, however, the results are disappointing, the composition range being small. However, it is thought that the results may have a certain interest in showing the relative accuracy obtainable in the measurement of refractive indices and optic axial angles. The usual determinative methods have been slightly varied. It is the purpose of this note to describe these variations and to compare the relative speed and scope of the method used with those obtained by other methods.

The lavas form a series at least 30,000 feet thick. The base is nowhere exposed. The olivines here described were obtained from only the lowest 4000 feet of this series. The dikes, with sills and small transgressive sheets, cut the lavas. They are well exposed in the steep cliffs for about 15 miles along the east coast, where they were collected. About sixty dikes outcrop in the 16 mile stretch of this coast, south of Ingia—the northernmost point of the island. Nearly all were sampled.

The rocks were crushed and sieved, those crystals being retained which had a diameter of approximately $\frac{1}{3}$ to $\frac{1}{2}$ mm. These crystals were then hand-picked under the microscope and a first selection made of olivines with clean-cut, straight, and moderately thin edges. After some trial it was decided to determine the index β, for the following reasons.

Crystals lying in such a position that one optic axis emerges almost vertically (thus giving β as refractive index) are easily recognized by their low interference-colours, which do not change on rotation. Such crystals can be easily and rapidly segregated, when confirmation of their

orientation is obtained by examination of their interference-figures. Sections yielding α or γ as indices cannot be recognized by their behaviour between crossed nicols, but only by determining their interference-figures. The necessity for this procedure makes their segregation a very much slower operation than in the former case, in which a large number of crystals can immediately be discarded during examination with nicols crossed. An alternative method consists in the manipulation of the crystals until the required orientation is produced. This is a difficult and tedious operation—especially so with olivine, which has no good cleavages. The 'statistical' method is also lengthy. In this instance γ and α are derived from the highest and lowest refractive index values, obtained by measuring a large number of random sections. It is, perhaps, not generally realized that this method can be applied to the determination of β. Thus, if γ' and α' be the higher and lower refractive index values obtained for each random section, γ' (max.) $= \gamma$, and α' (min.) $= \alpha$ (given a sufficient number of determinations). Also the mean of the lowest value obtained for γ' and the highest value obtained for $\alpha' = \beta$.

These 'statistical' methods, however, can properly be applied only to a set of crystals which possess exactly the same refractive indices. The results obtained give a mean value from a large number of measurements, whereas the method used in this determination shows small differences in refractive index occurring in olivines from the same rock.

The single dispersion method was used. Monochromatic light was obtained from a Tutton monochromator which had previously been calibrated by the spectra obtained from sodium, mercury, and cadmium lamps. This part of the method is precisely the same as that used by Deer and Wager [M.A. 7-447] for refractive index determinations on the olivines of east Greenland.

Since the refractive indices of these olivines were not higher than 1·74 it was possible to use a refractometer (Abbe-Pulfrich) in place of prism and goniometer for measuring the refractive indices of the liquids, which consisted of methylene iodide and α-monobromo-naphthalene in varying proportions.

Because of the comparative speed and simplicity of the refractometer method compared with the prism method, it was decided not to draw a large number of dispersion curves for mixtures covering the required refractive index range, but to plot four only. Special 'dispersion' paper (Hartmannsches Dispersionsnetz) was used for the plot. On this the curves closely approximated to parallel straight lines. The curve for any other mixture in this refractive index range was then rapidly obtainable,

by determining the refractive index of the mixture using the sodium lamp and then drawing a curve through the point so obtained on the Na[D] ordinate, parallel to the adjacent curve of the four already plotted.

A few drops of the mixture were placed on an ordinary microscope slide and small additions made of one or other of the component liquids until a match was obtained between the refractive index of the crystal and liquid at some point of the visible spectrum. A small portion of the drop was transferred in a capillary to the refractometer and its refractive index determined for the Na[D] wave-length. The dispersion curve for this mixture was then drawn as indicated above. On this curve was plotted the point which corresponded to the wave-length at which the match had been obtained (i.e. the wave-length at which refractive index of mixture = refractive index of crystal). The procedure was repeated for other mixtures and more points thus obtained on the dispersion curves of these mixtures.

The curve obtained by joining these several points is the dispersion curve for the olivine crystal. The point of its intersection with the Na[D] ordinate gives the refractive index [β] of the crystal for sodium-light.

Only those crystals were measured which showed the emergence of an optic axis close to the centre of the field—in no instance farther from the centre than half the radius. After a 'match' had been obtained between the refractive index of liquid and crystal it was found necessary to re-check the orientation of the latter to ensure that it had not moved during examination.

It is maintained that the procedure outlined above obviates, to a large extent, the difficult business of temperature control. Refractometer, slides, capillaries, and liquids are kept in the same room for at least twelve hours prior to the determinations, and the room-temperature is kept uniform as far as is possible. Immediately the refractive indices of liquid and crystal have been matched on the slide, a drop or two of the liquid is transferred to the glass hemisphere of the refractometer, and the refractive index at once determined. It seems reasonable to assume that any temperature variations which occur during this process must be very small. It is essential that the dispersion curve for the mixture should be obtained at the same time and at sensibly the same temperature as the refractive index 'match' between crystal and mixture.

The determinations of the optic axial angles were made on the universal stage, using rock sections. Monochromatic illumination—sodium lamp—was used. Owing to the large value of 2V, this angle could seldom

be measured direct, but was obtained from the position of one of the optic axes and a bisectrix.

Only those crystals were measured in which neither an optic axis nor the two measured optic planes made large angles—greater than 35° approximately—with the vertical. An exception to this was made, of course, in those rare instances where the crystal was so oriented that a bisectrix emerged nearly vertically, thus permitting a direct measurement of 2V. Five readings were taken of the position of an optic axis or optic plane, and the mean of these was used for plotting.

In order to obtain a reliable mean value for 2V, it was also found necessary to make the same number of measurements about γ as about α. A set of values obtained about one bisectrix usually showed fair mutual agreement, but differed by as much as 7° or 8° from another set obtained about the other bisectrix; whereas the mean of the two sets always showed good agreement with the value inferred from the measurement of β. In nearly every instance the mean of a set of angles, obtained about one bisectrix only, was found to depart farther from 90° than did the true value of 2V. No explanation can be suggested for this, but it is certainly not accidental, since it was of almost universal occurrence.

The results obtained are given in table I. The first column gives the number of the rock and indicates the field occurrence. The second shows the range in β for each set of crystals, which were measured in each rock. The number in parenthesis is the number of crystals in each set. In the third column is the value of 2V corresponding to the mean value obtained for β (from data by Winchell). The fourth gives the mean value of 2V, obtained by direct measurement from each set of crystals. The number in parenthesis shows the number of crystals in each set. The last column indicates the percentage range of Fe_2SiO_4 corresponding to the range obtained for β (column 2; from data by Winchell). β was determined for 63 crystals and 2V for 65, these totals being divided, as table I shows, between 10 rocks.

It has been stated that as great a reliance cannot be placed on 2V as on α or γ (and therefore, presumably, β also) for the determination of chemical composition of olivines. The results indicate the truth of this statement, if only a few crystals are measured, but show that the mean of a considerable number of determinations of 2V is as reliable a guide to composition (Fe_2SiO_4 percentage) as the mean value obtained for β—always provided, as stated above, that the same number of measurements are made about one bisectrix as about the other.

By reference to table I it will be seen that the mean value of 2V by

measurement (65 crystals) is 89·7° (+); whereas the value obtained by inference from β (63 crystals) is 89·8° (+). Although the mean values of a large number of measurements of β and 2V show excellent mutual agreement, individual coincidence is often not so good, and in these instances β is always more closely in accord than 2V with the composition (as determined from a considerable number of crystals). An illustration of this is furnished by dike no. D 14 (1) in which the variation in β is from 1·669 to 1·676 (corresponding to a range in composition from 9 to 12·1 mol. % Fe_2SiO_4). The variation in 2V, however, is from $83\frac{1}{2}°$ (+) to $87\frac{3}{4}°$ (−), corresponding to a composition range from 0 to 19 mol. % Fe_2SiO_4, which is probably considerably too great.

The refractive index variation (β) in some rocks (for different olivine crystals) is as large as 0·015—a composition difference of about 9 %—while in others the range is less than 0·003, i.e. within the limits of experimental error. Broadly the variation is greater in the olivines from the lavas than in those from the dikes. The former also contain a slightly higher Fe_2SiO_4 content (14·2 %) than the latter (11·8 %).

The highest value obtained for β in any crystal was 1·690 (19 % Fe_2SiO_4) and the lowest 1·666 (8·5 % Fe_2SiO_4). The data for 2V were 83° (+) and 84° (−), (0 % and 28 % Fe_2SiO_4) respectively.

TABLE I. Optical data of olivines.

Rock.	Refractive index variations (β).	2V, calculated from mean value of β.	2V, direct measurement on universal stage.	Molecular % Fe_2SiO_4 deduced from β.
Flow, no. 2	1·672–1·683 [6]	90·0° (±)	—	—
„ 22	1·675–1·690 [5]	89·2 (−)	89·0° (−) [8]	11·75–19·0
„ 25	1·676–1·684 [7]	89·75 (−)	89·3 (−) [6]	12·0 –15·25
„ 29	1·675–1·685 [5]	89·6 (−)	89·2 (−) [6]	11·5 –16·5
Dike, no. 2	1·671–1·683 [7]	90·0 (±)	89·75 (+) [10]	9·75–15·25
„ 14 (1)	1·669–1·676 [6]	89·0 (+)	88·8 (+) [6]	9·0 –12·1
„ 14 (2)	1·671–1·682 [7]	89·0 (+)	89·3 (+) [6]	9·75–15·0
„ 18 (1)	1·676–1·679 [7]	90·0 (±)	89·5 (−) [8]	12·75 (mean)[1]
„ 18 (2)	1·666–1·670 [5]	88·2 (+)	87·5 (+) [7]	8·5 (mean)[1]
Sill, no. 021	1·677–1·681 [8]	89·8 (−)	88·6 (+) [8]	13·9 (mean)[1]
		[63] crystals		[65] crystals

[1] Range in value of β here too small to indicate any composition differences.

Mean value of β in olivines from flows = 1·681
„ „ „ dikes = 1·675
Difference ... ... = 0·006 corresponding to a composition difference (mol. % Fe_2SiO_4) of 2·4 %.

The crystal-structure of violarite.

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[Read November 7, 1940.]

Waldemar Lindgren[1] described in 1924 a mineral present in nickel ores from southern Nevada which in colour, hardness, and stability differs from polydymite from Grüneau mine at Schutzbach, between Betzdorf and Daaden in Rhenish Prussia, and yet matches so-called ferriferous polydymite from Vermilion mine, Sudbury, Ontario. He proposed the name violarite both for the mineral from Nevada and for the 'polydymite' from Ontario on account of the violet-grey colour of the mineral on polished sections. Lindgren assumed that the iron found in the first analysis of 'polydymite' from Ontario was due to admixture with pentlandite and thence deduced nickel disulphide as the possible composition of violarite. In the light of subsequent results it is relevant to mention that Lindgren and Davy's work was based upon the study of polished ore sections but not in polarized light.

The more powerful method of studying polished sections in polarized light was applied to a large number of Sudbury ore specimens by M. N. Short and E. V. Shannon[2] in 1930. They concluded that although pentlandite is of common occurrence it never occurs associated with violarite in the same specimen. Violarite is intimately intergrown with chalcopyrite and the areas of violarite in a polished section are rimmed with pale-yellow millerite. The latter mineral had been mistaken by previous workers for pentlandite. Polished sections of both millerite and pentlandite have the same colour and only in polarized light can they be readily distinguished, millerite being strongly anisotropic and pentlandite isotropic. These and other observations led Short and Shannon to disagree with Lindgren and Davy on the paragenesis of violarite. Lindgren and Davy supposed violarite to be supergene replacing pentlandite, whereas Short and Shannon considered violarite to be hypogene and millerite to be supergene.

[1] W. Lindgren and W. M. Davy, Econ. Geol., 1924, vol. 19, p. 309. [M.A. 2–338.]

[2] M. N. Short and E. V. Shannon, Amer. Min., 1930, vol. 15, p. 1. [M.A. 4–335.]

Since violarite is isotropic and the closely associated chalcopyrite and millerite anisotropic, Short and Shannon were able to detach sufficient of the first mineral with a needle for chemical analysis. Their results yielded the formula $(Ni,Fe)_3S_4$ and they concluded that violarite belongs to the linnaeite group. F. W. Clarke and C. Catlett's[1] analysis of Ontario 'polydymite' showed less iron content, probably owing to admixed millerite. They were, however, correct in supposing that the essential constituent of the ore they analysed was a nickel iron sulphide.

Thus far the evidence taken in some detail from previous work suggests that violarite has a linnaeite rather than a pyrite type of chemical formula. Its isotropy in polarized light would, of course, fit either conclusion. My recent paper on bravoite from Derbyshire[2] draws attention to an upper limit to the nickel content of naturally occurring bravoite at 25 %. It is of interest to make quite certain whether violarite is a member of the pyrite-bravoite or of the linnaeite group. This can best be done by X-ray methods.

Through the kindness of Dr. A. L. Parsons the Mineral Department of the British Museum has recently received from Mr. E. A. Collins of the International Nickel Company of Canada, Ltd., a polished specimen of violarite detached from a mass collected by Dr. A. P. Coleman from the Vermilion mine. This specimen fits Short and Shannon's description exactly. A small fragment of the violarite was detached free from any adherent chalcopyrite or millerite. An X-ray photograph of the fragment revealed the lines corresponding to those of a face-centred cubic structure with edge 9·51 Å. X-ray photographs of linnaeite from Müsen, Westphalia, and of true polydymite from Grüneau mine show similar patterns both with smaller cell-edges, 9·40 and 9·47 Å respectively. These last two results are in fair agreement with those of G. Menzer[3] and those of W. F. de Jong and H. W. V. Willems.[4] Violarite, therefore, like linnaeite and polydymite, possesses the spinel type of crystal-structure with formula $(Ni,Fe)_3S_4$. This result confirms the optical and chemical work of Short and Shannon and refutes Lindgren's view.

Several specimens of so-called polydymite from Ontario which have been in the British Museum collections for some years have also been studied. Polished sections resemble exactly the above-mentioned violarite specimen and they all yield the same X-ray pattern. They are

[1] F. W. Clarke and C. Catlett, Amer. Journ. Sci., 1889, ser. 3, vol. 37, p. 372.
[2] F. A. Bannister, Min. Mag., 1940, vol. 25, p. 609.
[3] G. Menzer, Fortschr. Min. Krist. Petr., 1927, vol. 11, p. 315. [M.A. 3–339.]
[4] W. F. de Jong and H. W. V. Willems, Zeits. Anorg. Chem., 1927, vol. 161, p. 311.

undoubtedly violarite. It is a fortunate coincidence that the name polydymite can be retained only for the rarer mineral from Grüneau mine which exhibits well-developed octahedral, polysynthetic twin-crystals. Violarite shows no twin-lamellae on polished sections and the synonym ferriferous polydymite is therefore a less appropriate name.